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LEED STUDIES OF ADHESION TO A RHENIUM SURFACE

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ABSTRACT

Adhesion studies were conducted in a LEED apparatus with a quartz fiber contacting a rhenium $(10\bar{1}0)$ plane. Forces applied to the fiber in contact with the surface as well as those required to separate the solid surfaces were measured. LEED patterns were obtained before and after adhesive contact for both clean surfaces and surfaces with an oxygen layer. Adhesion of the quartz to rhenium occurred in the presence of, as well as the absence of, the oxygen layer. On surface separation, brittle fracture of quartz was observed to occur. Two structures were seen to develop with the admission of oxygen to the $(10\bar{1}0)$ surface of rhenium, a near (2×2) structure and also a (1×3) structure.

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SUMMARY

An investigation was conducted to determine the utility of LEED (low-energy electron diffraction) in the study of the process of adhesion. Experiments were conducted with a quartz fiber making adhesive contact to the $(10\bar{1}0)$ surface of rhenium. The rhenium surface was examined both clean and with a layer of oxygen present on the surface. The fiber was 1 millimeter in diameter and contact was made with the rhenium surface at loads from 20 to 160 milligrams and at temperatures from 20° to 800° C.

The results of the investigation indicate that LEED can be used to assist in understanding the adhesion process. In this study, it indicated that adhesion of surfaces will occur even in the presence of a surface layer of oxygen. Adhesion and separation of quartz from the rhenium surface results in brittle fracture of the quartz. With LEED the onset of plasticity or fracture can be determined. With adsorption of oxygen to the $(10\bar{1}0)$ surface of rhenium, two structures were observed, a near (2×2) and a (1×3).

INTRODUCTION

The adhesion behavior of materials in contact is extremely important to the field of lubrication. Based on the adhesion theory of friction, the friction coefficients measured are determined in part by the tendency of the two surfaces in contact to adhere. Further, one of the major modes of wear and probably the most significant in the production of failures in mechanical components is adhesive wear. While considerable research effort has been put forth to improve lubricants and thereby minimize adhesion, little has been done to gain a better understanding of the atomic nature of the adhesion process itself.

Many very useful tools have been developed in recent years to assist in the study of surfaces. Such devices as the field ion microscope, sterscan electron microscope, and LEED (low-energy electron diffraction) are some. LEED provides the capability, with some limitations, of establishing the atomic nature of a clean surface, the effect of surface films, and the extent of their coverage on a surface. It may also provide some insight into the nature and extent of surface changes produced by the adhesion process at the atomic level.

The objective of this report was to establish the usefulness of LEED in a study of the adhesion behavior of materials in solid contact. It is the intent of the report to show that LEED can lend to our fundamental understanding of the adhesion process. The materials examined to establish this usefulness were quartz in contact with rhenium metal.

Experiments were conducted with rhenium single crystals using a prism face $(10\bar{1}0)$. Rhenium was chosen for four reasons: (1) rhenium has a high modulus of elasticity, (2) its surface is relatively easy to clean compared to other refractories, (3) its surfaces have not been examined in LEED, and (4) the hexagonal metal rhenium has been shown to have very good friction and wear characteristics (refs. 1 and 2). All experiments reported herein were conducted with a 1-millimeter-diameter quartz flat-ended rod contacting the $(10\bar{1}0)$ face of rhenium. Loads covered a range of forces from 20 to 200 milligrams and rhenium specimen temperatures from 20° to 800° C. Studies were conducted with both clean and oxygen-containing surfaces.

MATERIALS

The rhenium single crystals examined in this investigation were made from zone-refined rhenium. The principal bulk impurities were 15 ppm tungsten, 5 ppm carbon, 4 ppm molybdenum, 3 ppm iron, 0.5 ppm oxygen, and the balance of the impurities were 1 ppm or less. The oxygen and hydrogen gases used in the experiments were reagent grade. The hydrogen gas was, prior to use, passed through a liquid-nitrogen-cooled molecular sieve to remove any trace amounts of contaminants.

SPECIMEN PREPARATION

The rhenium prismatic orientation was electric discharge machined from a 10-millimeter-diameter rod into slices 2 millimeters thick. It has been the practice to use thinner sections in LEED work. Thicker sections were selected here to minimize bulk recrystallization in specimen preparation (rhenium has a recrystallization temperature of 1400° C). After machining, the specimens were polished on papers down to 600 grit. They were then polished with diamond paste and finally electropolished with phosphoric acid. The crystals were electron-beam-welded to a polycrystalline rhenium holder. The face to be studied was reelectropolished in phosphoric acid and the orientation checked with Laue X-ray technique.

The quartz fibers were cut to size and then the surface was cleaned in an acid solution to remove any metallic surface contaminant.

APPARATUS

The apparatus used in these studies is shown schematically in figure 1. The single crystal surface mounted in the center of the chamber could be rotated 360°. This rotatability allowed for the making of adhesion measurements on the crystal surface shown in figure 1, then rotating the crystal 180° and obtaining a LEED pattern from the crystal surface in the adhesion contact area. The crystal could also be moved in the lateral and vertical directions.

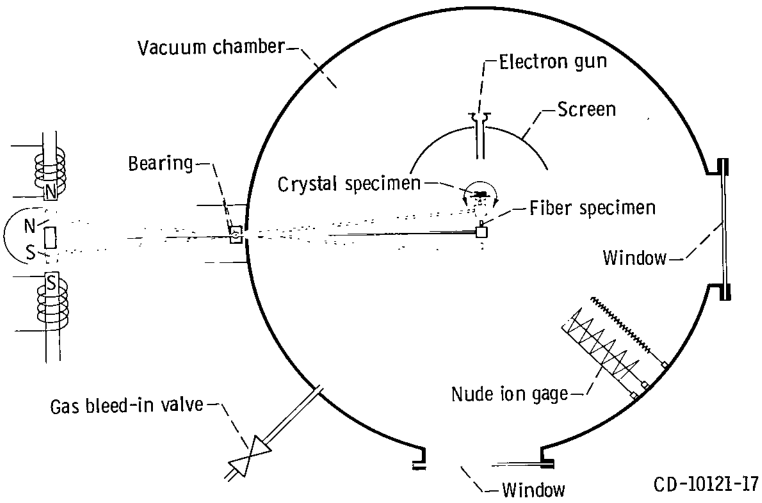


Figure 1. - Low energy electron diffraction LEED adhesion apparatus.

The crystal specimen was supported in the chamber by means of two rods which were insulated from the chamber and used to resistance heat the crystal. A 100-ampere ac power supply was used for resistance heating.

The quartz rod was cleaned by bringing the side of the rod (not the contacting tip) in touch contact with the rhenium crystal holder and heating. It was necessary to clean the quartz because of the strong bonding of water to quartz.

The fiber, which contacted the single-crystal metal surface, was mounted in a stainless-steel holder which, in turn, was mounted to a 1.5-millimeter-diameter stainless-steel beam. The beam was mounted in a bearing-containing yoke. At the end of the beam beyond the pivot point, and opposite the fiber specimen, was a small permanent magnet. Outside the chamber wall were two electromagnets. The permanent magnet and electromagnets were positioned in such a manner as to have like poles facing each other. A simple variation in the current applied to the electromagnets could be used to move the beam. The current applied to the electromagnets was calibrated in terms of the force applied in the adhesion experiments. Load applied to the surfaces in contact and the force required to separate the crystal surfaces were then measured by current.

The LEED electron optics and the vacuum system were of the standard type used by those engaged in LEED studies and are adequately described in the literature (ref. 3). The basic LEED system was obtained commercially. The electron optics was of the varian three-grid type. The beam diameter at the crystal surfaces was approximately 0.6 millimeter, thus enabling the entire LEED pattern to be taken within the 1.0-millimeter-diameter contact area. The vacuum system consisted of vacsorb pumps, an ion pump, and a sublimation pump. The system pressure was measured with a nude ion gage and all experiments were conducted with the vacuum system in the range of pressures from 8.0×10^{-11} to 2.0×10^{-10} torr. No cryopumping was used.

RESULTS AND DISCUSSION

The prismatic plane $(10\bar{1}0)$ of rhenium was examined in these studies. The plane and the surface unit mesh (smallest structure which represents the whole surface) are shown in figure 2. The unit mesh is similar to that for a (112) surface in the body-centered-cubic system.

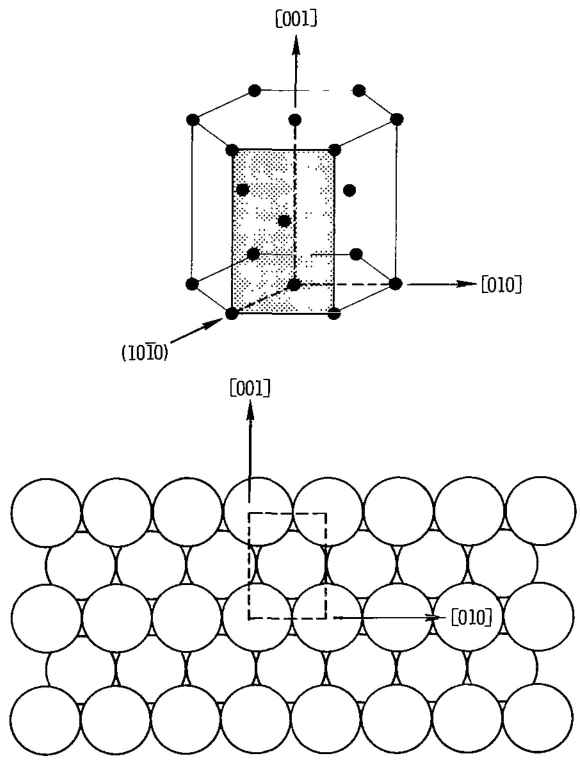
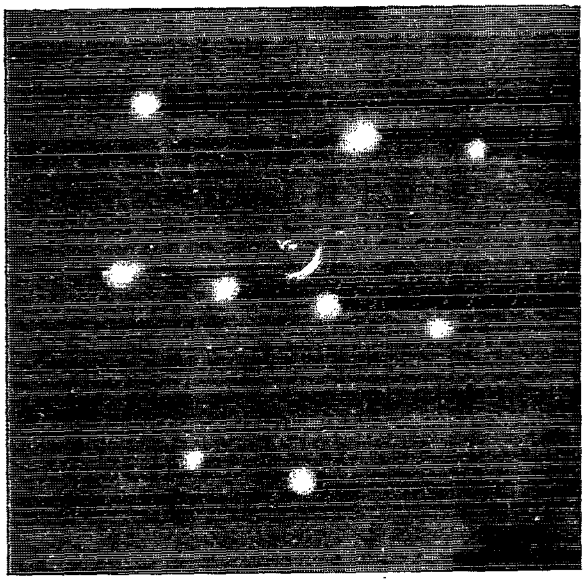
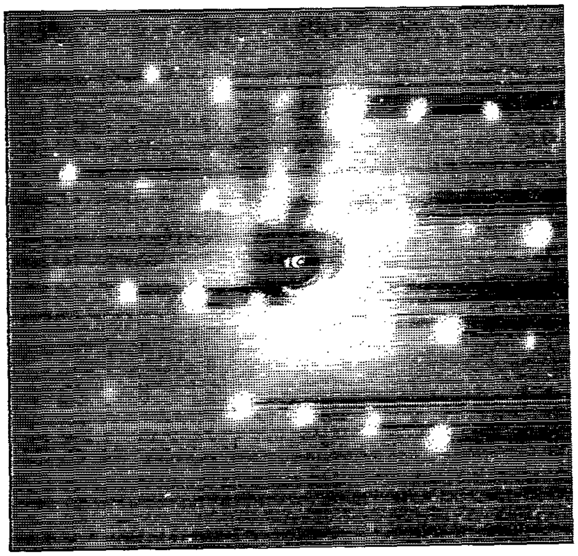


Figure 2. - Atomic arrangement of $(10\bar{1}0)$ rhenium surface with unit mesh.

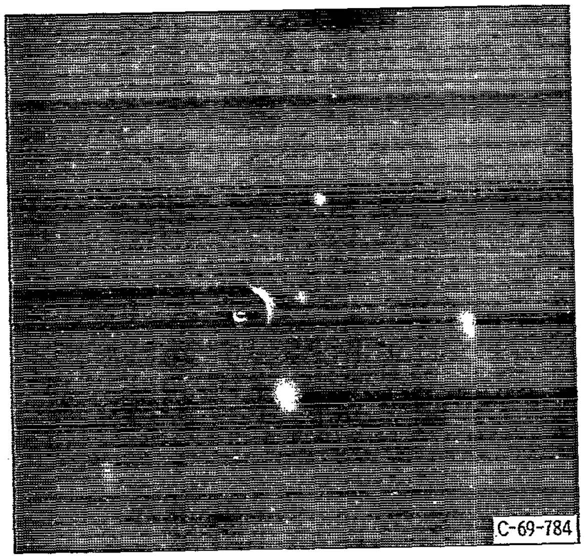
LEED patterns were obtained from the prismatic surface shown in the ball model of figure 2. The surface, prior to obtaining the patterns, was heated to 1000° C for 3 hours and then exposed to 5.0×10^{-8} torr-second of hydrogen at 5.0×10^{-7} torr, and then the pressure was reduced to 1.0×10^{-10} torr and the crystal heated to 1180° C. It was held at 1180° C for 3 hours. The resulting LEED patterns, after this surface treatment, are presented in figure 3. Note the rectangular pattern characteristic of the unit mesh in figures 3(a) and (b). After having obtained the patterns of figures 3(a) and (b), the crystal



(a) Clean surface; beam voltage, 60 volts.



(b) Clean surface; beam voltage, 175 volts.



(c) Contaminated surface of crystal after exposure at 1.0×10^{-10} torr for 72 hours; beam voltage, 60 volts.

Figure 3. - $(10\bar{1}0)$ Surface of rhenium as shown by low energy electron diffraction (LEED).

was allowed to stand in the vacuum chamber over a weekend. The pattern of figure 3(c) was then obtained. Figures 3(a) and (b) represent a clean surface at two beam voltages, while figure 3(c) shows the same surface after 72 hours exposure to the residual gases in a vacuum chamber in the pressure region of 1.0×10^{-10} torr.

While the surfaces shown in figures 3(a) and (b) are believed to reflect a clean rhenium surface and a few years ago would have been held to be so, this today cannot be said with certainty. It is always possible to have patches of amorphous surface contaminant present. The advent of Auger emission coupled with LEED can assist in the identification of such surface species (ref. 4).

The objective of this report, as stated earlier, is to use LEED as a tool in the study of adhesion. Adhesion experiments were therefore conducted with a quartz fiber contacting the rhenium surface of figures 3(a) and (b) under various conditions. Quartz was selected as the contacting surface because (1) it is, in the form used, amorphous and therefore would not give a superimposed pattern over rhenium, (2) it could serve as a source of oxygen for the surface, and (3) it fractures in a brittle manner.

The force of adhesion of quartz to the $(10\bar{1}0)$ surface of rhenium was measured for various contact times under a load of 20 milligrams. The results obtained in these experiments are presented in figure 4. An examination of figure 4 indicates that the force

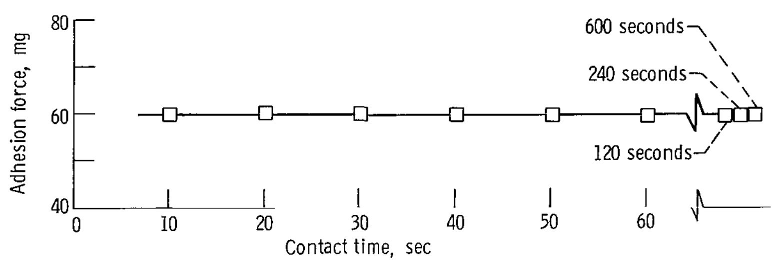
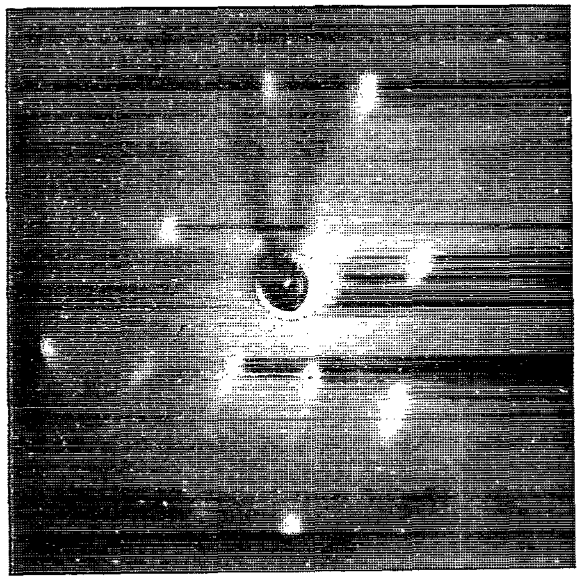


Figure 4. - Force of adhesion for 1-millimeter-diameter quartz fiber in contact with clean $(10\bar{1}0)$ rhenium crystal surface. Specimen temperature, 20° C; contact load, 20 milligrams.

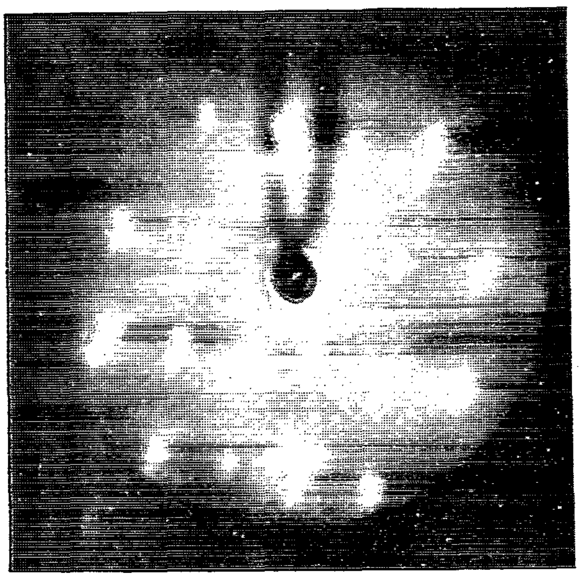
of adhesion was 60 milligrams and was independent of contact time. At the higher loads of reference 5, a marked dependence of adhesion as a function of contact time was found for metals. This dependence (in ref. 5) was believed to be due to creep; under the loads used, the surfaces were thought to be in the region of plastic flow.

In figure 4, the independence of adhesion to contact time indicates that the contacts at the interface are elastic. While adhesion occurs between the quartz and the rhenium, the load is sufficiently light such that the elastic limit of quartz has not yet been reached. Evidence for this is shown in the photograph of figure 5(a) taken after the 20-milligram adhesion experiments. If this LEED pattern is compared to that of figure 3(a), little change on the surface appears to have occurred. Figure 5(a) would indicate that essen-

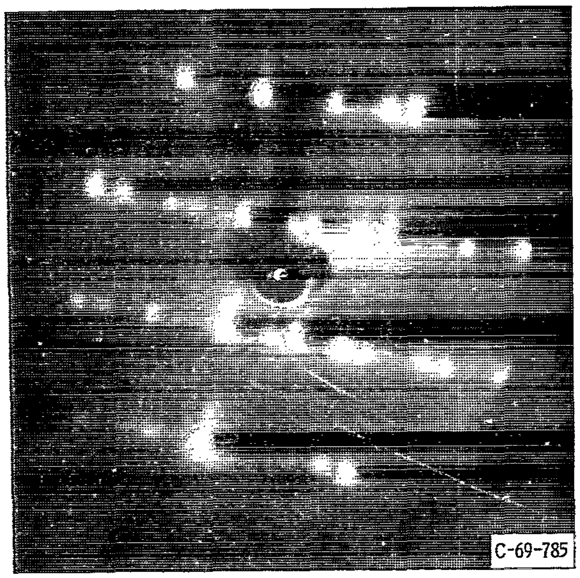
(a) Load, 20 milligrams; beam voltage, 60 volts.



(b) Load, 120 milligrams; beam voltage, 60 volts.



(c) Load, 120 milligrams; beam voltage, 105 volts.



(d) Load, 120 milligrams on surface exposed to oxygen for 5 minutes at 10^{-6} torr; beam voltage, 135 volts.

Figure 5. - $(10\bar{1}0)$ Surface of rhenium as shown by low energy electron diffraction (LEED) after adhesion experiments with quartz fiber.

tially no surface damage occurred to the rhenium and little, if any, oxygen or quartz remained adhered to the rhenium.

It might be anticipated that, at some load, the elastic limit of the lower elastic modulus material of the adhesion couple (quartz) would be exceeded. If oxygen bonding to rhenium occurred, and the strength of this bond were greater than the fracture strength of the quartz, brittle fracture would occur in the quartz. Experiments were, therefore, conducted with the adhesion contact made at various loads. The results of these experiments are presented in figure 6.

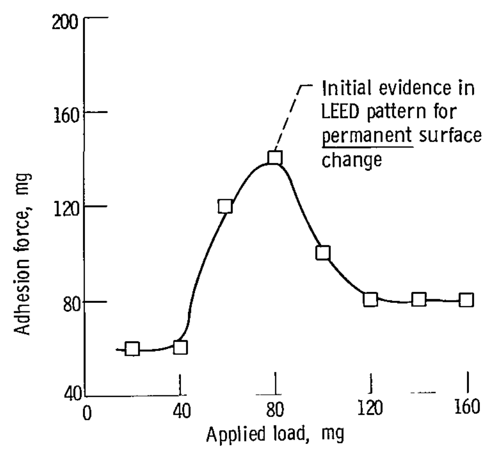


Figure 6. - Force of adhesion as function of applied load for 1-millimeter-diameter quartz fiber in contact with $(10\bar{1}0)$ surface of rhenium. Specimen temperature, 20° C; contact time, 10 seconds.

The data of figure 6 indicate the influence of the transition from elastic to brittle behavior on the part of the quartz. At loads of 20 and 40 milligrams, the contact was elastic with no evidence of any permanent change taking place to the rhenium surface as evidenced by LEED patterns of the surface. At 60 and 80 milligrams, however, a marked increase in adhesive force was noted. This increase is believed to reflect a very strong oxygen bonding to the rhenium surface and an exceeding of the elastic limit of the quartz resulting in brittle fracture of the quartz in tension. Thus, the chemical bonding of oxygen to rhenium was stronger than the bond for fracture of the quartz; hence, fracture occurred in the quartz. If this were so, then a change in the rhenium surface as seen by LEED should occur. Examination of LEED patterns at various loads revealed a permanent change in the surface at loads in excess of 80 milligrams.

It is hypothesized in reference to figure 6 that, at 20 and 40 milligrams, because of elastic recovery and bond "peeling," the contact area in tension is equivalent on the removal of the load and just prior to the application of tensile force for both load conditions.

This would account for the equivalence in adhesive force. At 60 to 80 milligrams, sufficient interfacial bonding has occurred that the quartz must be fractured in tension. This leaves quartz on the rhenium surface as has been shown. Here the force required to separate the interface has become greater than the force for brittle fracture in the quartz. In the load region from 100 to 160 milligrams, considerable fracture of quartz occurs on loading simply because the elastic limit has been exceeded, and for brittle materials, this means fracture. This would then reduce the force required to separate surfaces. The failure to observe an increase in adhesion force between 100 and 160 milligrams reflects brittle, rather than ductile, behavior.

Such changes are shown in figures 5(b) and (c). The pattern has now become streaked, with spots being elongated. This streaking could have resulted from one of three sources: (1) deformation of the rhenium, (2) the presence of oxygen on the surface, and (3) the presence of quartz on the surface.

The streaking of the pattern of figures 5(b) and (c) could not be accounted for by the plastic deformation of the rhenium. It has six times the elastic modulus of quartz. Heating the crystal surface to 1000° C resulted in no surface rearrangement, thus it could not be due to the presence of elemental oxygen. The patterns of figures 5(b) and (c) were, therefore, due to quartz adhered to the rhenium surface. This conclusion was substantiated by microscopic examination of the surface. The photomicrograph of figure 7 shows the points of real contact within the apparent contact area and the presence of quartz at these sites.

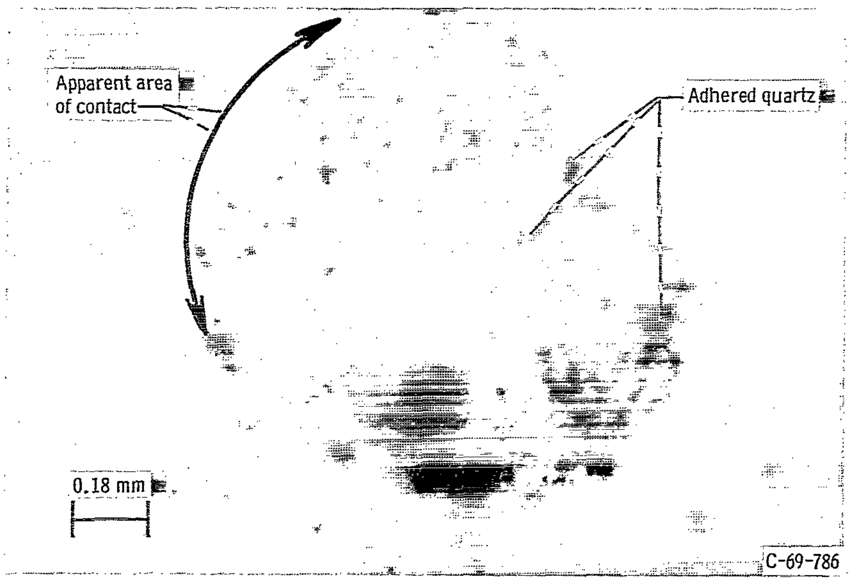


Figure 7. - Contact area on rhenium (10$\bar{1}$0) surface after adhesion experiment with quartz fiber at 150-milligram load.

The brittle fracture of a material such as quartz can be described in terms of the Griffith theory for brittle fracture where existing flaws can be postulated in the brittle material (ref. 6). In an experiment such as that described herein, there can be no doubt that flaws exist near the interface. The critical stress for fracture, which would be reflected in the adhesive force, can be represented by the Griffith equation as

$$\sigma_c = \left(\frac{2SE}{\pi C}\right)^{1/2}$$

where

σ_c critical stress to fracture

S surface energy per unit area

E modulus of elasticity

C 1/2 flaw or crack length

It can be seen from the equation that as the the crack length increases, the stress necessary to propogate it decreases. This results from a decrease in potential energy with crack growth. While such considerations might be very significant in bulk tensile studies, in adhesion where the tensile test is micro in nature and the maximum crack length achievable before fracture is so small, the changes in fracture stress may be so small as to be of second-order importance.

It might be expected that temperature would have some effect on the adhesion behavior of materials. Adhesion experiments were therefore conducted at various temperatures, and the data obtained are presented in figure 8. At temperatures to 600° C no change in adhesion force with temperature was noted. At 700° and 800° C an increase was observed. This increase may reflect the beginning of the effects of quartz softening.

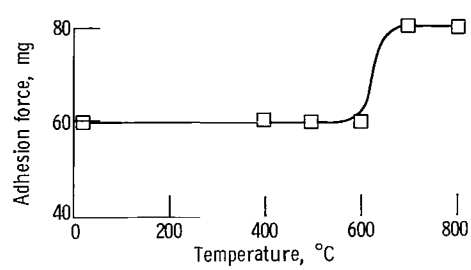
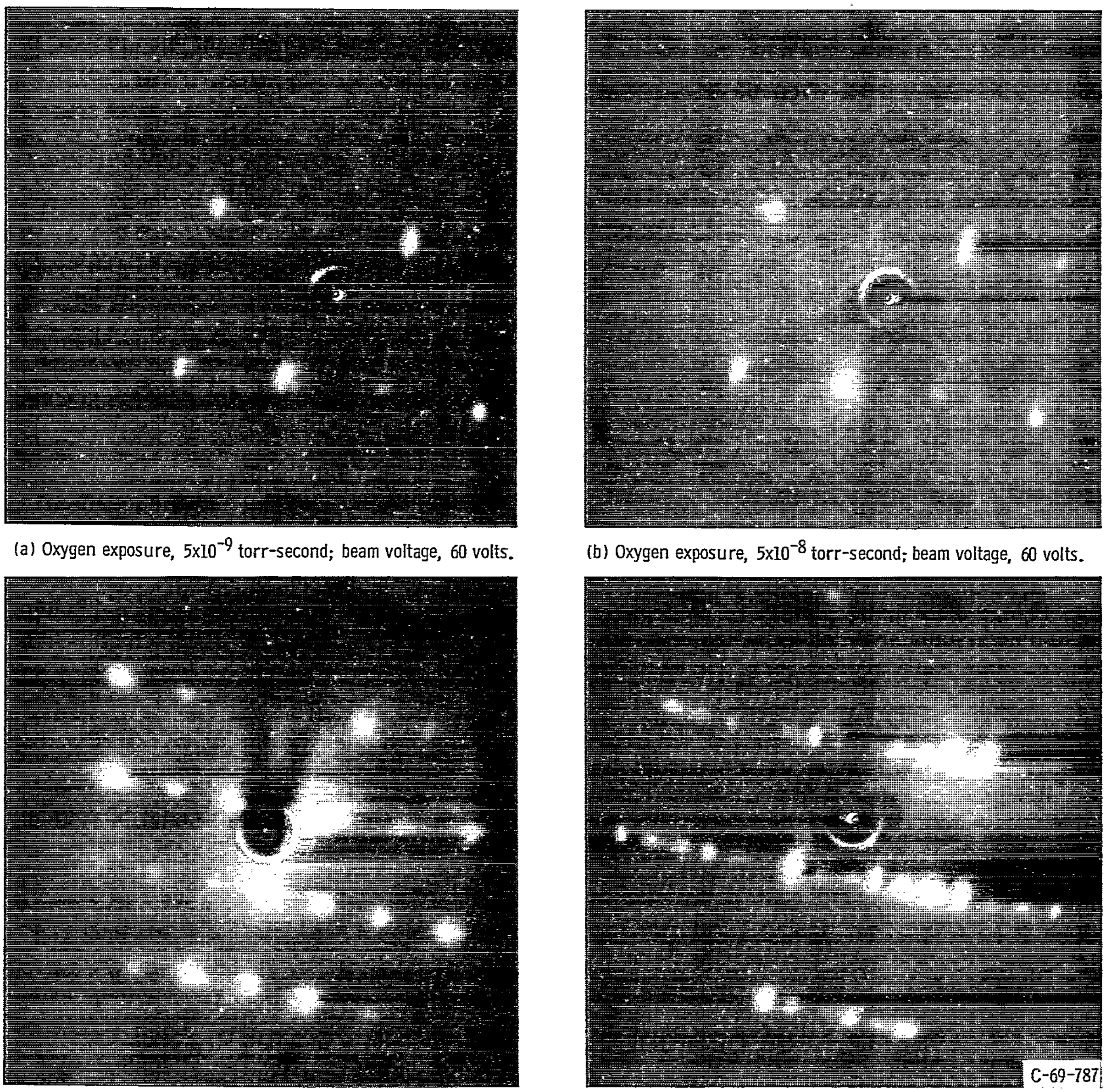


Figure 8. - Adhesion force as function of temperature for 1-millimeter-diameter quartz fiber contacting $(10\bar{1}0)$ surface of rhenium. Applied load, 20 milligrams; contact time, 10 seconds.



(a) Oxygen exposure, 5×10^{-9} torr-second; beam voltage, 60 volts.

(b) Oxygen exposure, 5×10^{-8} torr-second; beam voltage, 60 volts.

(c) Oxygen exposure, 5×10^{-8} torr-second after heating to 800° C; beam voltage, 60 volts; near (2x2) structure.

(d) Oxygen exposure, 5 minutes 10^{-6} torr after heating to 800° C; beam voltage, 85 volts; (1x3) structure.

Figure 9. - $(10\bar{1}0)$ Surface of rhenium as shown by low energy electron diffraction (LEED) with various oxygen exposures.

In lubrication studies, metal surfaces are normally covered with surface oxides. In order to determine the influence of surface oxygen on the adhesion behavior of rhenium, experiments were conducted with surfaces exposed to various amounts of oxygen.

The manner in which oxygen became attached to the surface can be seen in the LEED photographs in figure 9. The random arrangement of oxygen on the surface resulted in an obscuring of the rhenium pattern. This can be seen by contrasting figures 9(a) and (b) with figure 3(b). In figure 9(c) an oxygen exposure of 5×10^{-8} torr-second resulted in the development of a near (2×2) structure after heating to 800° C. Further exposure to oxygen and subsequent heating to 800° C resulted in the oxygen developing in a (1×3) structure on the rhenium surface. The two structures developed can be compared with the primitive (1×1) structure of the clean surface in figure 3.

For those unfamiliar with the surface descriptions commonly used in LEED, the near (2×2) structure, for example, means in reference to the Miller indexing of surfaces that new spots have appeared in the pattern at the h/2 and k/2 positions in the diffraction pattern. The appearance of these new spots in the diffraction pattern implies that the chemisorbed oxygen atoms are arranged in a structure that has dimensions that are twice those of the rhenium substrate in both the [001] and [010] directions. It thus becomes obvious that for the (1×3) structure, the oxygen is arranged in the [001] direction in the same dimensions as the rhenium atoms while in the [010] the dimensions are three times that of the rhenium.

Adhesion experiments were conducted on the (1×3) surface structure of figure 9(d). The experiments were conducted at various loads and the results obtained are presented in figure 10. The curve from figure 6 is included for reference purposes. The first and most obvious result is that adhesion of quartz to rhenium will occur even in the presence

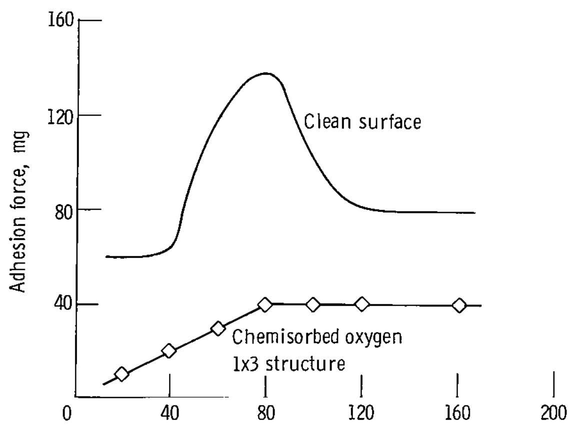


Figure 10. - Adhesion force as function of applied load for 1-millimeter-diameter quartz rod contacting $(10\bar{1}0)$ rhenium with oxygen present on surface. Specimen temperature, 20° C; contact time, 10 seconds.

of chemisorbed oxygen. It must be borne in mind that this oxygen layer is not the thick oxide normally encountered in oxidized surfaces but probably, based on the measurements of reference 3 with tungsten, represents on the order of a monolayer. Under load, then, it is assumed from the data of figure 10 that rhenium to quartz contact must occur through the oxygen chemisorbed layer to account for the adhesive forces measured.

In figure 10 the adhesive force increases with load to 80 milligrams; beyond that load it remains relatively unchanged. This latter region was earlier referred to as the region in which fracture of the quartz occurred.

The LEED surface pattern obtained after an adhesion experiment at 120 milligrams load is shown back in figure 5(d). While spot distortion occurred, it was nowhere near as severe as was observed in figure 5(c) under the same load but in the absence of the chemisorbed oxygen.

SUPPLEMENTAL DISCUSSION

It is felt that, since most of the data obtained in this report depend upon the fracture characteristics of quartz (silica), some discussion of its fracture behavior is warranted. Most amorphous materials are thought of as being brittle, and upon exceeding their elastic limit, they will fail in a brittle manner. This has been found not to be true; plastic flow has been observed, for example, in soft glasses (refs. 6 to 10). In fact, with soft glasses under high hydrostatic pressures, gross plasticity has been observed (refs. 7 and 8).

The picture is, however, quite different for silica and a few other very hard glasses. Here it is clear from data in the literature that flow cannot occur below the material's intrinsic strength. Elastic strains achievable are so high in a material such as silica that elastic behavior must be considered to exist to fracture (ref. 10). In silica the structure is, however, relatively open and subject to compaction at high stress levels. With such a material, though, plastic flow has not yet been observed in deformation experiments.

From the data presented in this investigation, it is clear that, when clean quartz is brought into contact with clean rhemium, adhesion will occur. It is proposed that this bonding is oxygen-to-rhenium chemical bonding. A mechanism for such bonding is proposed and ahown in the schematic of figure 11.

In silicon dioxide, silicon is surrounded by four tetrahedrally bonded oxygen. When one considers a very large molecule composed of these tetrahedrally bonded silicon ions, the overall stoichiometry is such that the SiO_2 formula results. If two of these tetrahedrally oxygen-bonded silicon ions are taken as a dimer, the structure on the top of figure 11(a) is what is observed in a plan view. There is a layer of oxygen, then silicon, and the silicon is covered by another layer of oxygen.

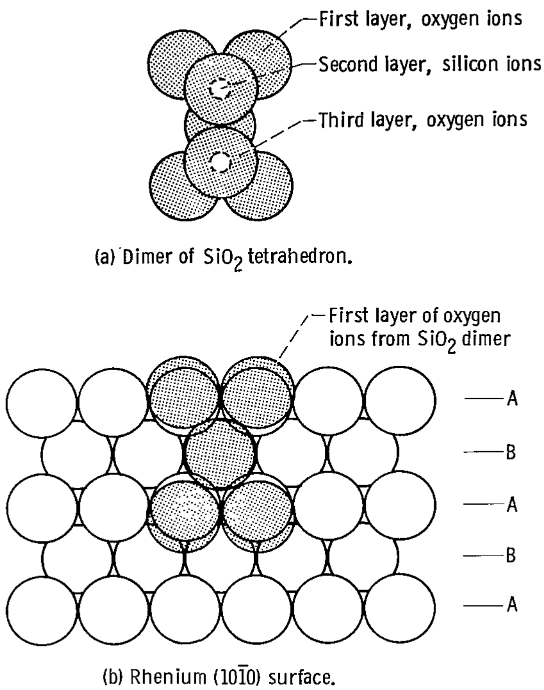


Figure 11. - Possible surface sites for bonding of silicon dioxide to rhenium (10$\bar{1}$0) surface.

When the ionic size factors and bond distances in quartz and the atomic size factor and arrangement for rhenium are considered, the interfacial match is that which is seen in figure 11(b). The match is good for oxygen bonding to the rhenium A rows of figure 11(b) in the [010] direction.

The oxygen of the quartz would almost, of necessity, be bonded in figure 11(b) to the A rows of rhenium atoms because these rows comprise the outermost layer and insufficient interaction of oxygen with B rows could take place because of A row hinderance. Good evidence for oxygen bonding to A rows of rhenium atoms is found in the adsorption of oxygen to rhenium. In figure 9(d), oxygen bonding was observed in the A rows. It is thought, therefore, that quartz in adhesion experiments bonds in a similar manner.

Metal-to-oxygen bonding of other oxides has been observed. In reference 11, it was observed with various metals in contact with aluminum oxide in vacuum wetting studies. Further, it was observed to occur in adhesion and friction experiments with various metals in contact with aluminum oxide in reference 12.

CONCLUSIONS

Based upon the LEED and adhesion measurements made in this investigation with rhenium, the following conclusions are made:

1. LEED (low-energy electron diffraction) can be effectively utilized in the study of adhesion.

a. The onset of material transfer (quartz to rhenium in this study) was readily discernible with the aid of LEED.

b. The nature of the surface with which adhesion experiments were made could be defined.

2. Adhesion of quartz to rhenium was found to occur even in the presence of a chemisorbed oxygen layer.

3. The adhesion behavior of a brittle solid to a metal is markedly different than for metals in contact (even for those metals that are normally considered brittle).

4. With respect to LEED itself, the $(10\bar{1}0)$ surface of a hexagonal metal has been observed with LEED, and oxygen was observed to develop structures on this surface. In this investigation, both a near (2×2) and a (1×3) structure have been observed; oxygen structures on rhenium were observed.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, February 26, 1969,
129-03-13-09-22.

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